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**Patentanmeldung Nr.    Patent application No.    Demande de brevet n°**

02292769.3

Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
p.o.

**R C van Dijk**



Anmeldung Nr.:  
Application no.: 02292769.3  
Demande no:

Anmeldetag:  
Date of filing: 06.11.02  
Date de dépôt:

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
If no title is shown please refer to the description.  
Si aucun titre n'est indiqué se référer à la description.)

Method of adhesion measurement at the interface between layers

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)  
revendiquée(s)  
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/  
Classification internationale des brevets:

G01N19/00

Am Anmeldetag benannte Vertragstaaten/Contracting states designated at date of  
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR IE IT LI LU MC NL PT SE SK TR

"METHOD OF ADHESION MEASUREMENT AT THE INTERFACE BETWEEN LAYERS"

**Field of the Invention**

The present invention relates to a method of measurement of adhesion strength and, more particularly, to a method of measurement of adhesion strength at the interface between layers in a multi-layer stack, especially in a multi-material stack.

**Background Art**

Microelectronic devices comprising integrated circuits may comprise stacks of layers. There are many situations in which it is desirable to measure the adhesion strength between two layers of material. Typically, this is a requirement during manufacturing processes of said microelectronic devices, when testing products for reliability. Often the two layers will consist of different materials, but it can also arise that there is an interface between two layers made of substantially the same material.

During manufacture of such microelectronic devices comprising integrated circuits, it is desirable to measure the adhesion strength between layers in a wafer, notably layers deposited as thin films. Ideally this would be done at various locations across the surface of the wafer. Wafer-testing techniques used to date involving stress-measurement techniques such as the curvature method, or x-ray measurement are described in the publication entitled "Intrinsic stress in chemical vapour deposited diamond films: An analytical model for the plastic deformation of the Si substrate" by J.H. Jeong et al, in the Journal of Applied Physics, 90(3), pp 1227-1236, 1<sup>st</sup> August 2001. However, these techniques only provide information about residual stress in the wafer in general, they do not provide a measurement of adhesion strength between the different layers in the wafer at specific locations on the wafer.

In other fields, various measurement techniques are known which do allow adhesion-strength to be determined. Tests including stress measurement using a microcantilever are known from the publication entitled "Quantitative surface stress measurement using a microcantilever" by M. Godin et al, in Applied Physics Letters, 79(4), pp 551-553, 23<sup>rd</sup> July 2001. Methods that also

for the critical energy. Calculation of the adhesion strength uses this estimated radius value.

There are disadvantages, too, with the method described in US 5,838,446. Notably, ablation of materials is likely to generate dust, which will be undesirable in many manufacturing environments, e.g. in the microelectronics industry. Moreover, this technique requires use of a particular sample geometry which involves handling the sample to an extent which may be undesirable (e.g. if the sample is a semiconductor wafer being used to manufacture microelectronics devices). Further, in order to calculate a single adhesion strength value, measurements must be taken at a number of locations on the sample and the resultant data combined – leading to undue complication and a relatively lengthy calculation time.

US patent 5,438,402 describes a laser spallation technique for determining tensile strength at the interface between a substrate and a coating. In such laser spallation techniques, a mechanical impulse is applied to the substrate and coating. In order for the mechanical impulse to be transmitted to the substrate/coating, it is necessary to provide an energy-absorbing coating on the free surface of the substrate (the surface remote from the coating), and a confinement plate on the energy-absorbing layer. A pulse from a laser is used as the energy source in this arrangement. Adhesion strength is calculated based on the movement of the free surface of the coating.

It is an object of the present invention to provide a new technique for evaluating adhesion strength at an interface between two layers, for example between layers of two different materials in a multi-material stack.

Preferred embodiments of the present invention provide an adhesion-strength evaluation method which is simpler to implement than the methods known heretofore. More particularly, in these preferred embodiments, a value for adhesion strength can be calculated without the need to process data relating to a set of locations on the sample or the need to use a complicated confinement structure.

Preferred embodiments of the invention allow adhesion strength to be measured at discrete locations over a surface.

### Detailed description of the Embodiments

The stress caused by a laser shock applied to a material depends upon the wavelength and the energy of the laser pulse used to produce the shock. More particularly, the higher the wavelength and energy of the laser pulse, the greater the stress that is caused. The behaviour of a surface undergoing a laser shock is described in more detail in "Electron diffusion in metals studied by picosecond ultrasonics" by G. Tas et al, appearing in Physical Review B, 46(21), pp15 046- 15055, 1<sup>st</sup> June 1994.

A laser pulse of appropriate wavelength and energy, impacting directly on a layer of material, can create sufficient stress at the interface between that layer and another to which it is bonded so as to overcome the forces of adhesion between those two layers. (This rupture of the interface between the two layers can be detected, for example using an acoustic sensor or x-ray reflection device. The adhesion strength between the two layers can thus be determined by applying laser pulses of increasing wavelength and/or energy directly to a sample undergoing test, noting the wavelength and energy of the laser pulse which just causes sufficient stresses to provoke rupture of the interface, and calculating a value for adhesion strength based on these critical values of wavelength and energy.

A laser shock can apply a pressure of up to 300 MPa or more which is generally adequate to crack the interface between two layers of material. For example, the adhesion strength of the interface between a layer of Ni and a layer of  $\text{Si}_3\text{N}_4$  is approximately 100 MPa; the adhesion strength of the interface between a layer of Al and a layer of  $\text{Si}_3\text{N}_4$  is approximately 100 MPa; and the adhesion strength of the interface between a layer of Nb and a layer of  $\text{Al}_2\text{O}_3$  is approximately 300 MPa. Thus, the technique of the present invention can effectively be applied for measurement of adhesion strength.

A preferred embodiment of method of measuring adhesion strength between two layers, according to the present invention, will now be described with reference to Fig.1. In this example, the adhesion strength is being measured at the interface between two layers in a stack which consists only of those two layers. However, the interface under test may be between the two

layers 1, 2 to separate, in other words the interface 3 cracks. This event creates a sound, the sound waves propagate through the second layer 2 (as illustrated by the black ovals in Fig.1(b)) and can be detected, for example, using the acoustic sensor AS, which outputs a signal O. The adhesion strength ( $\sigma_{1,2}$ ) between the layers 1, 2 is a function of these values of wavelength and energy ( $\lambda_2, \epsilon_2$ ). More particularly, the pressure, P, at the surface of the sample can be determined from the following equation:

$$P = 0.622A^{7/16}Z^{-9/16}\lambda^{-1/4}\tau^{-1/8}I^{3/4},$$

where A is the atomic weight of the layer 1, Z is the ionisation degree of the plasma,  $\lambda$  is the wavelength of the laser,  $\tau$  is the pulse duration and I is the maximum power density of the plasma. (Incidentally, in the case where the laser shock wave traverses more than one layer before reaching the interface undergoing test, the above equation is applied to each layer traversed).

In a case where a colouring laser is used, it is preferred to set the wavelength of the laser pulse to a first value,  $\lambda_1$ , and the pulse energy to a first value  $\epsilon_1$ , before applying the first pulse to the sample at a given location. If the resulting laser shock is insufficient to cause debonding at the interface 3, then the pulse energy is increased by an increment  $\Delta\epsilon$ , and a second pulse is applied to the sample at the same point. The method continues, increasing the laser pulse energy in steps until either debonding occurs at the interface 3 or the maximum possible pulse energy for the laser is reached. If the maximum pulse energy is reached before debonding occurs, then the wavelength of the laser is increased by an increment, the pulse energy returned to its lowest value, and the process repeated at the new wavelength. Eventually, the wavelength ( $\lambda_2$ ) and energy ( $\epsilon_2$ ) values necessary to produce debonding will be reached.

In a case where a single-wavelength laser is used, the testing process preferably consists in setting the pulse energy at an initial value  $\epsilon'_i$  for the first pulse applied to the sample, then ramping up the energy value by an increment  $\Delta\epsilon'$  for each subsequent laser pulse, until debonding is detected. Preferably, a laser sensor LS (e.g. a photo-diode) is provided to detect laser light exiting

the sample under test, it may be preferred to start with a pulse energy which is already close to the maximum and/or with a wavelength which is not the lowest wavelength of the laser. Moreover, the size of the increment in energy and/or wavelength may be set differently depending upon the nature of the material(s)  
5 at the interface undergoing test.

Any reference sign in a claim should not be construed as limiting the claim.

## CLAIMS:

1. A method of measuring adhesion strength between first (1) and second (2) layers of material in a stack of two or more layers, the first and second  
5 layers (1,2) being in contact at an interface (3), the method comprising steps of:  
applying a plurality of laser shocks directly to a free surface of said stack of layers by causing a plurality of laser pulses of respective different wavelength and/or energy to impact said free surface,  
detecting cracking of the interface (3) on application of one of said  
10 plurality of laser pulses;  
determining the wavelength and energy of the applied laser pulse causing cracking of the interface (3); and  
calculating a value for adhesion strength of the first and second layers (1,2) based upon the determined wavelength and energy values.  
15
2. The adhesion-strength measurement method of Claim 1, wherein the laser shock application step comprises applying said plurality of laser pulses at the same location on the free surface of said stack until cracking of the interface is detected .  
20
3. The adhesion-strength measurement method of any previous claim, wherein the detecting step comprises detecting cracking using an acoustic sensor.
- 25 4. The adhesion-strength measurement method of any previous claim, wherein said first and second layers are layers of a semiconductor wafer product.
5. The adhesion-strength measurement method of any previous claim,  
30 wherein said first layer (1) is at one end of the said stack and a surface of said first layer (1) constitutes the free surface of the stack on which the laser pulses impact.

Measurement of adhesion strength in a multi-material stackAbstract of the Disclosure

The strength of adhesion between two layers (1,2) is evaluated by applying a series of laser shocks directly to the surface of one (1) of the layers.

5 Adhesion strength is determined based on the wavelength ( $\lambda_2$ ) and energy ( $\epsilon_2$ ) of the laser pulse ( $L_2$ ) creating the shock which causes rupture of the interface (3) between the two layers (1,2).

(Fig.1(b))

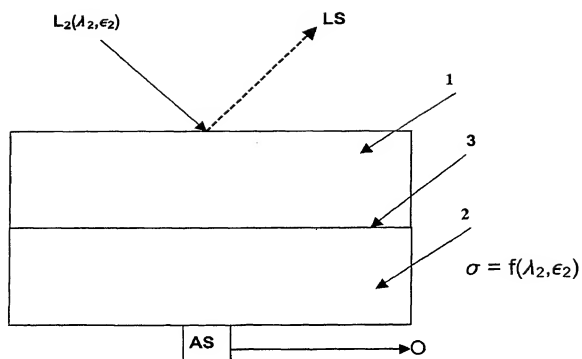


FIG.1B

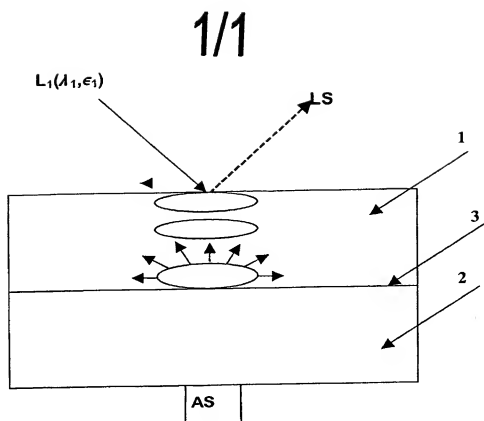


FIG. 1A

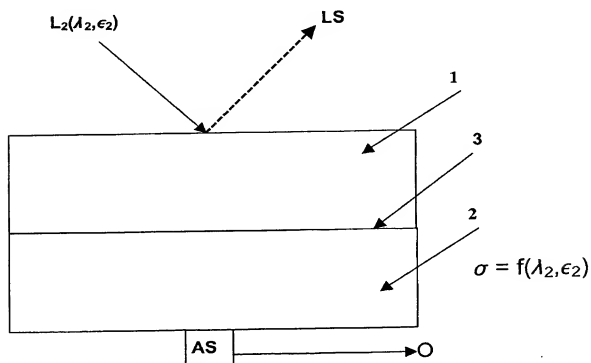


FIG. 1B